Basic Beamline Operations

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0. Introduction

This manual describes how to perform the most common tasks on the beamline. These include steering the beam, setting the spot size, navigating on the sample, mapping, and taking EXAFS spectra. This manual does not go into beamline tuneup, maintenance or data analysis. Also, diffraction isn't covered, as this requires changing the mechanical setup in the sample area and doing some additional work with the detector. A plan is afoot for improving this situation.

If you are not familiar with LabVIEW controls and, especially, graphs, read the document on Common LabVIEW Conventions before proceeding. The beamline programs are all written in LabVIEW and make extensive use of graphs, cursors, array indicators and the like.

There are cheat-sheets for the mapping and EXAFS programs. You may want to print these out for quick reference. If you have problems, refer to the Troubleshooting guide, a shortcut to which is on the Desktop of the data analysis computer (probably where you're reading this). A copy is appended to this writeup, however the one on the Desktop may be more current.

There is also a note describing the beamline optics. Read this to understand the role of the various mirrors and the roll slits. This note will be replaced with a full-length paper, to be submitted to *J. Synchrotron Radiation*, about the beamline.

Tricky points which are not obvious and which can be confusing and troublesome are written in *italics*. Names of controls and indicators in the programs are in Arial, as they are on the actual screens on which they appear.

I. Getting beam

You should have been taught how to get in and out of the hutch and how to get beam, but here's a quick review: To lock the hutch and get beam, start by pushing the small black search button on the upstream wall of the hutch next to the optics box. Exit, and while holding the hutch door shut, turn the key CCW and withdraw it. Put the key in the lock on the PSS panel (there's a sticky with the message 'use this key' where the key goes) and turn CW. An annoying noise will start. The shutter will be enabled when the noise stops. The shutter switch is on the panel to your left, over the label 'BR1032-06'. This switch has a locking handle, so pull gently and move the switch to the OPEN position. You can do this before the noise stops, but the shutter won't open until the appropriate time.

To get back into the hutch, close the shutter and wait for the red 'SAFETY SHUTTER CLOSED/KEY RELEASE OK' light to come on. While pushing the black button under this light, turn the key CCW and withdraw it. Put it in the lock on the hutch and turn to unlock the hutch.

The shutter is open when the green light over the shutter switch is on. Below the shutter switch is a small viewport. You should be able to see a purple glow if the beam is actually on.

The next step is to steer the beam onto the roll slits. This may need to be done at the top of a fill, or when trying to get a small probe spot. Once done, the beam seems to hold for most of a fill, and sometimes for days.

The relevant controls are on the beamline-control computer. The right-hand keyboard, monitor and mouse is connected to two different computers through a 2-port KVM switch. The desktop for the data-taking computer (`UXASES`) has many icons on a light-blue background. That for the control computer (`1032_BL_CONTROLS`) has fewer icons on a dark-blue background. Steering the beam, setting the spot size, and changing the I_0 and I_t gains are done with the control computer. To switch between computers, hit the `Control` key twice.

To steer the beam, start by moving the roll-slit sizes to 20x10µm. This is done using the Motor Monitor program, which shows as a window about 85mm high by 95mm long. First, set the horizontal size. Click on the Motor control (bottom left) and select Horizontal Slit Size. Type '20' into the Goal window (top center) and hit the Move button (top left). The slit will close through zero and come back to 20µm. Similarly, set the Vertical Slit Size to 10µm.

Next, find the diagnostic PIN diode. There is a plexiglas cage which holds the roll-slit assembly (big cross with gears and motors). Downstream of this is a 2-3/4" cross with a black linear-motion feedthrough on top. This feedthrough has an indicator showing the position of the PIN diode. In normal operation, this should be all the way up. Use the crank-handle and locking screw to move the actuator so that its indicator shows at the position marked IN. The PIN diode now intercepts the beam. You will

probably see a flash of light in the window when you do this; this is just a piece of YAG attached to the stick on which the PIN diode rides.

The output of the PIN diode may be read on the middle one of the row of three DVMs above the shutter switch. If you don't see anything, try power-cycling the current amp to which the PIN diode is connected (should be on top of the Bruker AXS half-rack; trace cables if you can't find it). Now, go back to Motor Monitor and select M1 roll. Make sure the Jog Size (just above the Motor control) is set to 0.02; type this in if it isn't. Now, use the Jog buttons (left side) to maximize the reading from the PIN diode. This reading is echoed as Channel 2 in the set of readouts on the right side of the large Beamline 10.3.2 Beamline Control System window, so you don't need to crane your neck reading the DVM. There is a backlash move when you hit the upper jog button, so wait for the motion to stop before doing anything else. Next, maximize the reading using the M1 Tilt motor with a jog size of 0.0005.

You have now steered the beam through the roll-slits. Lift the PIN diode out of the way (as high as it goes) and set the slit sizes to what you want. Here are some rough guidelines for slit sizes:

Slit size HxV, µm	Spot size HxV, µm	
500x100	16x7	full size, max flux
100x100	7x7	
50x20	5x5	smallest beam

In some cases, slitting down a little, say to 300x50µm on the roll slits, can improve stability and normalization by cutting off the fringes of the beam.

The gain on the I_0 and maybe I_t detectors should be set at this point. This is done using the Keithley 428v2.VI window, which has a tan background. The top half is for I_0

and the bottom for I_t. They both work the same way. To change the gain, click and drag on the pointer in the knob. The numbers (3-10) are the exponent in the gain expressed in V/A. Thus, 1nA of I_0 signal will yield a reading of 1 on the 9 setting. *The setting will not 'take' unless you then hit the blue* Set *button below the* Gain *knob*. The maximum reading which can be recorded is 10V, so the gain should be set so that the signal is less than 10. Note that the signal increases twofold between the bottom of a fill and the top of the next fill, so plan your setting to avoid saturating the readout if you're going to cross a fill.

This concludes the list of things you have to do on the `1032_BL_CONTROLS` computer. Hit `Control` twice to switch back to `UXASES`, where the data-taking programs live.

It may occasionally be necessary to check the monochromator 'tweak', that is the parallelism of the crystals. This is adjusted as follows: Find the black Picomotor driver (so labeled). It has a knob on the front with 8 positions. Switch it to 2. There is a hand-paddle with three sliders connected to this unit. Gently work the A slider to maximize beam. If you want to detune, use this control to get whatever degree of detuning you want. You can use the MCA utility (*v.i.*) to see when the harmonics go away.

II. Sample mounting and positioning

The standard sample mount is a trapezoid with one 'bent' side. A drawing of this, with dimensions, may be found on the beamline website at http://xraysweb.lbl.gov/uxas/Beamline/Hardware/Hardware.htm. The shortest side is the bottom. If you hold it with the short side down and the 'bent' side to the left, you are looking at the front, or upstream face. Something like a microscope slide may be

attached to the back side so that the surface of interest shows through the 1" hole in the front. The sample may stick out from the front surface as the stage can be moved backwards from the focus, but the point of interest (POI) should not be any farther back than flush with the back surface of the holder. We want the POI to be at the focus, which is rather close to the end of the I_0 chamber, and if the POI is too far back, the sample stage and holder may bump into the I_0 chamber. It is more common for samples to be attached to the front of the holder. For small samples or bits of material such as loose soil or plant matter, you can stretch kapton tape over the hole in the holder and stick the sample there. Try to avoid wrinkles in the tape as that causes parallax offsets in the beam position and also lets the sample creep. For more-solid samples, I find that aluminum tape holds with less sample motion than kapton. However, this tape is full of Fe, Mn, and Cu, so you need to keep it well away from your sample if you're looking for these elements. We also have an insert for TEM grids.

Note that the standard sample mount and the arm it rides on are not suitable for diffraction. For that, we replace the sample arm with another one which is not as good mechanically, but allows the scattered beams to be seen by the CCD.

The standard sample holder is held by three slotted pins on the sample arm. Two of these pins engage the long straight side, while one engages the bent side. This geometry is a magnified version of the one on 7.3.3. The block with the three pins is on a pivot and may be lowered to a horizontal position by loosening a screw with a red plastic head. Tighten this before attempting to move the sample around.

The whole sample-stage assembly rides on a manually-controlled leadscrew translator which moves the sample towards or away from the optics box. This motion is

provided so that the POI on the sample may be brought to the plane of the focus. Getting this positioning right is important for two reasons: the depth of focus is only about 1mm, and the sample microscope does not look straight along the beam, thus leading to a parallax offset between where you think the beam is hitting and where it really is.

An overhead view of the sample area is shown in Figure 1a, with the sample stage shown with the front of the sample at the focus (solid lines) and behind focus (dotted lines). The difference between these positions lies in the adjustment of the longitudinal stage. What you want to do is to get the place where the beam hits the sample to lie on the focal plane. Since this plane is invisible, it's not obvious how to do that. Now, the fact that the sample microscope doesn't look straight along the beam can be used to advantage. The crosshairs on the video monitor (right screen in hutch; big one outside) are set up so that they line up on the beam when the sample is in focus. Thus, if the sample is out of focus, the beam will hit to the left or right of where the crosshair shows. This situation is shown in Figure 1b, in which the monitor is shown as it would appear with the sample in the same planes as shown in Figure 1a. In both cases, the beam is hitting the center of the black circular grain.

To adjust the focus correctly, we first make a rough adjustment of the longitudinal position to get the image in the sample microscope to be in sharp focus on the monitor, then find a feature with X-rays and move the longitudinal slide so that this feature is on the crosshairs. *Move the longitudinal stage only, not the crosshairs*, which are your reference to where the sample plane is.

III. Moving, mapping and MCA

Now we need to be able to move the sample around and see what's being hit by the X-rays. The programs you will need for all operations other than what was described in Section I reside on the data-taking (UXASES) computer. Switch to it, if you're not already there, by hitting Control twice.

We first need to move the sample. The Manual Stage program (Set Stage Position, according to its window) may be accessed two ways: You can get it from its shortcut on the desktop, or by pushing the Manual Stage button in the XY Mapping program. Let us use the latter method, as we will need to do some mapping. First, invoke the mapping program, if it's not already running, by using the desktop shortcut. The program will come up, but in a state in which it's not really running. There will be a small arrow in the upper left corner of the window which is white in this state and black if the program is actually running. If the arrow is white, click on it, and the program should start up. At this point, you can push the Manual Stage Control button which should bring up the manual stage program, running. There are four buttons in a diamond pattern at the upper left of this program, and a control labeled StepSize(um). These are the jog controls. When you use these, remember that they move the sample, not the crosshair. You can use these controls to move around on the sample, looking for your POIs.

To understand what and where the X-rays are actually hitting, use the MCA utility. There is a separate manual for this, so I'll just go over the basics. The program is invoked from a desktop shortcut and needs to be started with the little arrow in the upper left. After a few seconds, it starts collecting spectra. Use the graph tools to scale it as you wish. By default, the abscissa is in channels, for which the conversion to energy is

10eV/channel. Thus, the Fe K_a line is at 640 channels. The highest-energy large peak is usually the elastic, at whatever energy you've set for the monochromator. You can use the MCA display to find a feature on the sample which corresponds to some feature in the X-ray yield. Typically, you can find the edge of a grain visually and with the MCA. Use a vertical edge to get the best accuracy in finding the right point. You can also use the edge of Al tape, but that tape is thick enough to cause significant parallax error. In some cases, you can't be sure of how the signals you see with the MCA correspond with the features you see visually. In that case, you may have to do a coarse map to know what's what.

A screen shot of the mapping program is shown in Figure 2. Use this Figure to follow along with the description of the steps in mapping. The basic steps are:

1. If needed, do an I_0 offset. This is to make normalization work and isn't really required for navigation. This step is only needed if you have changed the I_0 gain or have not done an offset since the mapping program was started. To do this, close the shutter and push the Measure I0 offset button. *This offset is separate from the one in the EXAFS program. Doing one does not keep you from having to do the other.*

2. Set the incident energy where you want it. Some suggested energies:

Purpose	Energy
General	10keV
Lighter elements	5-6keV
As	12900
Pb	13050
Br (trace)	14500

To set the energy:

a) If the EXAFS program is running (v.i.) click on the Operations tab, enter the desired energy in the Target energy window, then click Move.

b) Otherwise, switch back to 1032_BL_CONTROLS and use Motor

Monitor to move Mono eV to the desired value in the same way as you would set a slit size.

3. Define your MCA bins (same as Regions of Interest). This is done by entering the appropriate data into the Regions of Interest Settings controls (called out in Figure 2 as MCA Bins). Unlike 10.3.1, this system only collects ROIs, not complete spectra at each pixel, so if you didn't ask for it, you can't get it from the data. For each ROI, you can enter a name, which gets entered into the file header, and the upper and lower limits for the bin, in eV. These numbers are 10 times the channel numbers you'd pick out of the MCA Utility. For most users, most of the time, the default ROIs are good enough. If you want to add a bin, simply fill in one of the grayed-out ones at the end, *then stop the program (red STOP button) and restart it*. The change won't 'take' unless you start and stop. You don't lose any information by doing that. You can change the data for an existing bin without stopping and starting.
4. Define the starting and stopping points in your map. The map is always a rectangle whose upper left corner is the Start point and whose lower right is Stop. To define a new Start, move to where you want the map to start, then hit the Get Start Position button in the middle of the manual stage window. Defining a Stop position works similarly. If you want to center a map about a point, you can go there, then use the jog controls to move by some amount to the upper left, get the start position, then move twice that much to the lower right and get stop. *The program ignores the first and last three points in each line of data as these are taken during acceleration and deceleration, so the map will be narrower than you expect*.

5. Hit the RETURN button on the manual stage program, then push the Scan Params button in the mapping program. There are three tabs, Stage, File and I0. Start with the Stage tab. The numbers on white backgrounds are the ones you can set; the ones on gray are derived or fixed. The top row includes the pixel size in the X and Y directions and the dwell time. A typical fast map will have 20x20µm pixels and a 50ms dwell time. The estimated time for the complete map is shown at the bottom of the window. It's easy to miss the Hours indicator, so look for that. Leave the Settling time and # of pts to clip from each end alone. The next tab is the File tab. It lets you set the title of the map (written into the file header), the name and the directory. Make sure you've set it to your current data directory. The Scan number auto-increments on each scan unless you set it back, thus helping prevent overwriting. Leave the Extension at xrf. The final tab is I0. This sets the value of I_0 below which a beam dump is declared. You can leave it at 0.05.

5. Pull up the manual stage program and hit the Move to Start button. Return from the manual stage program, then push the Run button on the mapping program. A map should start. If it gets to the end and a Bad Data indicator lights up, and that keeps happening, then something is mechanically wrong. The stage may be

hitting something, something may be loose, or you may have to slow down the acquisition by increasing the dwell time or decreasing the X pixel size.

The Abort button stops a scan, but not until the stage has reached the end of a line. If you do that, remember to move to the start position before trying again.

There are several ways to move to a specific point on the sample. The simplest is to use the jog buttons. However, if the mapping program is up, you can use the Move to position button on the bottom of the manual stage screen to move to where the cursor is on the map. This is useful for zeroing in on a feature of interest. There are also position registers. These are like 'bookmarks' for positions. You can load them, name them and move to any one of them. They don't actually do anything except hold positions for later reference. You can load one either by using the Load position button or by filling in the coordinates by hand. Of course, you can also Move to start and Move to stop.

IV. EXAFS

The point of all this mapping is often to acquire EXAFS or XANES spectra of interesting spots. We therefore turn to the EXAFS program. The first thing that needs to be done is to find the spot of interest. This is often a small area rich in the element of interest. If the area is small, then the EXAFS data will be better if you sit exactly on the spot where the count rate is maximized, even if there are plenty of counts. The reason is that if you're a little off and there's any beam or sample motion, this motion will be reflected in the signal, causing noise or strange backgrounds. By sitting on a maximum of yield, you cancel out, to first order, the effects of motions.

To find the spot, set the beam size as appropriate and move the mono to an appropriate energy, say 200eV above the edge. Use the MCA program to look at the signal in the peak corresponding to the element you want. Set an MCA ROI (see MCA utility manual) so you can see the count rate in counts/second. Now use the jog controls in the manual stage program to move around and maximize yield. With the spot size set large, you should finish up with steps of no more than 2μm for a localized spot. Diffuse spots can be examined with bigger step sizes. For a small beam, you should get down to 0.5-1μm motions. Now you're sitting on the spot.

The detector has limits on how fast it can count. The total counts in all channels (0-2047, usually the 1st bin set in the MCA utility) should not exceed 300,000/second. If it does, you can do one of the following:

1. Filter using Al foil. There are usually some pre-made sheets which go in a little slot at the snout of the detector. This is good when most of the counts are due to an interfering element whose fluorescence occurs at a lower energy than that you're interested in. The canonical example here is Zn EXAFS in soils, which are full of Fe and Mn.

2. Pull the detector back. The detector is on a slide. Clockwise on the shiny wheel pulls it back.

3. Slit down. This is preferred where the sample is vulnerable to radiation damage as you then want to use your incident flux in the most efficient way.

The EXAFS program has several complicated and non-intuitive aspects. One of these is that for historical reasons it's called mainscreen1. Another is the scaler map file. The program is designed to count any of a wide variety of inputs, including analog inputs, a counter board, and the Ge detector. The scaler map file tells it what physical devices to associate with a logical scaler. For basic EXAFS, in which you count all 7

elements together in one channel, we have a number of pre-made scaler map files. There is a shortcut on the desktop for the scaler map file in use, and a number of shortcuts for scaler map files for different elements to be counted. These files are all alike except for the specification of which MCA channels to count. Thus, the files for Fe and Zn look like this:

Fe	Zn
[Gate] board=1 counter=0 [Scaler 0] type=660x counter=1 board=1 [Scaler 1] type=660x counter=3 board=1 [Scaler 2] type=XIA detector=-1 **roilow=605** **roihigh=671** [Scaler 3] type=XIA detector=-1 roilow=0 roihigh=2047 [Scaler 4] type=end	[Gate] board=1 counter=0 [Scaler 0] type=660x counter=1 board=1 [Scaler 1] type=660x counter=3 board=1 [Scaler 2] type=XIA detector=-1 **roilow=835** **roihigh=893** [Scaler 3] type=XIA detector=-1 roilow=0 roihigh=2047 [Scaler 4] type=end

The entries in bold are the ones which differ from element to element. These files specify that the first and second scalers (Scalers 0,1) are counters. In practice, these are always connected to I_0 and I_t, respectively. The third scaler is the fluorescence channel. The `detector=-1` entry shows that all 7 elements are summed. The `roilow` and `roihigh` entries show the limits of the MCA bin used. The entry for Scaler 3 looks like the one for Scaler 2 except that it encompasses all channels. This is used for deadtime correction. The final entry isn't actually a scaler; it just indicates the end of the list.

Scaler maps are described in more detail in another writeup in the `10.3.2 Docs` folder.

To set the scaler map, you have to stop the EXAFS program (using the big red STOP button, not the tiny red stop-sign icon), edit the scaler map, then start mainscreen1 again. This action throws out the scan parameters and you have to remake or reload them. That's why you are offered the opportunity to save the scan parameters when you exit the program.

The scan parameters are what tell the program what energies to work at, how many points to take, how long to count, how many regions, where to write the data, etc. The easiest way to generate one of these is to copy it from a previous file (often somebody else's) and edit it. The file is a text file, so you can edit it off-line, but it's easier to use the scan editor built into the EXAFS program to do it. In that spirit, Figures 3a-3e show screen shots of the tabs available when editing a typical scan file. This file shows a scan on the Zn edge with 6 regions of energy defined. The first is a pre-edge region and is tabulated at a coarse 5eV/step. The next is the XANES region, at 1eV per step. After that follow EXAFS regions in which the step is constant at 2eV but the count time goes up linearly with energy. The settling time is the time allotted for the mono to settle down after each point and is always 0.5sec. There is enough overhead per point that it's not worth going below 2sec/point.

Figure 3b shows the file-related stuff. The final path name, shown on a blue background, is the concatenation of the directory name, the base file name, the scan number (in 3-digit format) and the extension, which should always be `dat` so that the analysis programs can read it. The scan number is auto-incremented in each scan.

Figure 3c shows the dump-detection criterion. The criterion is that the counts in the selected scaler, with no offset removed, must be greater than a user-define factor times the offset of that scaler. Otherwise, a beam dump is declared. Thus, the dump factor should be set slightly greater than 1. This offset may be read from an indicator on the right side of the Operations page. This 'factor' approach was done so that the user almost never has to change this setting. In the example shown in Figure 3c, the factor is 1.2, and the offset is 6228, so the minimum counts in I0 below which a beam dump is called is 1.2x6228=7474. Normally, you don't have to do anything to it, but if you read in an old scan file, then check it and change it to 1.2 or whatever your favorite number is.

The Set tab of the scan edit screen tells the program how many scans to take. There is a fairly complex option for automatically moving the stage between scans, but for the purposes of this basic manual, I'll leave that out. Thus, the switch at the bottom of this screen is left in the No - stage stays put position. The estimated time for the set to complete is shown on a blue background.

Finally, the Plot tab indicates what should be plotted by default. The plotting may be changed on the fly at any time during or after a scan, but this tab affects what default plotting specification is written into the file. In the example shown, we are plotting the third scaler (Scaler 2 - fluorescence) divided by the first (Scaler 0 - I_0) without taking the log of the ratio. See the manual for the EXAFS Data Editor for more detail on plotting specifications.

Anywhere an energy is required, you can enter it in a number of ways. Here are some examples of each of the different modes:

8976.73	8976.73eV
cuk	Cu K-edge energy

cukf	Cu $K_{\alpha 1,2}$ fluorescence energy
cuk+200	200eV above Cu K edge
cuk+200/3	(Cu K-edge energy+200)/3
present	Where it is now
start	Start of scan
mid	Middle of scan
end	End of scan

Thus, it is never necessary to look up the edge energies and do arithmetic. This notation is also useful for such things as moving to mid-scan.

Some useful things to know about scan definitions:

1. You can change the time-per-point in mid-scan. This may be useful for speeding up a scan to get it in before the fill.

2. If you want the current scan to finish, but don't want to do more, you can go into the Set tab and change the number of scans to 1.

3. If a beam dump occurs, the program will terminate and save the present scan, then, when beam comes back, start a new scan with the scan number incremented by 2 (a bug). It will attempt to take the full quota of good scans you programmed in the Set tab.

At this point, you're almost ready to take EXAFS. In fact, you are ready for XANES. The problem is as follows: The monochromator keeps the beam height constant on the K-B mirrors as a function of energy by moving the second crystal. This motion is quantized in half-micron steps. Sometimes, for reasons unknown, these steps will show up as non-normalizing features in the data, 80eV apart at the Zn edge, for instance. These features are subtle enough not to cause problems for XANES, only EXAFS. Thus, we need to freeze that motion during EXAFS scanning. This is done using the monochromator servo switch. This switch is the left-most of a row of four on a black box which is found behind the beam pipe and under the rack which is labeled BR1032-4. This box has cable with a big connector coming out the upper right and the

switches on the upper left, labeled Closed/Open Loop Select. For EXAFS mode, move to mid-scan with the switch down (enter mid in the Target energy field and hit the Move Mono button) and then flip the left-most switch to the up position. Don't touch any of the other three.

When moving to a distant energy (>1keV away), flip this switch back down to the normal position or you will lose beam.

Another 'gotcha' is the counter offset. *Redo this if you change gains or are starting the EXAFS program after having quit out of LabVIEW.* Close the shutter and hit the Measure offset button on the Operation page. Look at the four numbers in the pink-backgrounded indicator on the right. The third and fourth should be small (<100). If they are large, do it again - the detector electronics had a hiccup.

To recap, here's the checklist for starting EXAFS:

1. I_0 gain. Is the gain set right to keep from saturating or having too little signal? If you're at very low or very high energy, is the gas appropriate? N_2 is OK for most energies.

2. Scaler map. This only needs to be changed if you change edges.

3. Scan definition. Check the scan range, dump level, and filenames.

4. Offsets. Need to redo them?

5. Mono servo switch. Is the switch up? Did you change edges since the last time you did the move-to-mid/switch up ritual?

6. Spot size. Appropriate for the size of the feature? Beam motions and other mechanical noise seems to be worse when the spot size is comparable to the feature size, so you may either want to flood or put a pinpoint beam on the feature, depending on its size. You have to try it to see what will work best for your particular sample.

7. Sample position. Did it drift since you started doing all this other stuff?

8. Count rate. Is the detector at the right distance, with the right filtering to

keep the total count rate below 300,000 while preserving as many good counts as possible?

If you're truly ready, hit the Start button, sit back and watch. It is probably easier to find out by experiment what the controls on the Plot page do than to read about it. However, I will note that the plot only updates on each data point when a scan is in progress.

The smaller plot below the main one is used for inspecting I_0. It consists of I_0, but with a smooth (cubic) background divided out and the log taken, so you can better see glitches and other such features. For instance, if a glitch represents a 1% drop, it will show up as a dip of 0.01 depth.

If you want to do XANES, there's another important step - calibration. The EXAFS program's plot screen offers a Differentiate switch, which is useful for locating the edge of your standard. The standard is often a foil run in transmission. For transmission, we usually use a PIN diode, except when the Bragg glitches from that diode are too severe, in which case we use an ion chamber. To use the foil, you can tape it onto a sample holder, mount it in the usual way, then turn the holder using the knob on top until the foil is flat on to the beam. There is an Allen-head setscrew which is used to unlock this degree of freedom. Alternatively, you can swing the sample holder out of the way and use some Newport bits to hold the sample in place. The PIN diode is good enough for calibration XANES; you don't have to bother with the ion chamber. Run a XANES scan in one region around the edge and use the Differentiate function to see where the edge is. You can use the cursor to get a number. Once the scan is done, move to that energy, then type in the nominal value for that edge energy (e.g. cuk for Cu), then

hit the tiny green Reset Mono button under the Target energy field. You should now be calibrated. The calibration holds for days at a time.

If you have a number of points on which you want to do EXAFS and you want to get some sleep, it's possible to program the system to take EXAFS spectra at each of a set of programmed positions. This is done using the options exposed when you flip the Use Positioning? switch in the Set tab of the scan editor, as shown in Figure 4. The current position appears on the left. An array holding positions and repeat counts appears towards the right. For each position which appears in the array, the program will take a number of scans specified by the Repeat count on the right. Thus, in this example, the first, third and fourth positions are to be done once, and the second twice.

To set up a position, first go to it using the Manual Stage VI. Usually, this is done with reference to a map, tweaked up by looking for a local maximum in fluorescence yield. Use the index spinner on the upper left of the Positions indicator to scroll to the entry you want to modify or add. Push the round green button labeled Load current pos'n. Repeat for all positions you want to run.

To go to any of these positions, use the index spinner to make the one you want to go to appear at the top of the list, then hit the square green button Go to this pos'n. This will move the stage to that spot.

Now, the number of scans which will be done is still controlled by the # scans in set control at the top right. Thus, you have to make sure to tell it to do as many scans as you want. You usually want a full round of all the specified positions. The number of scans in such a set is given by the Length of cycle indicator, which has a blue background and is under the arrow to the right of the Load current pos'n button.

There is an important bit of logic which must be mentioned. The program decides what position to go to by looking at the scan number of the file it's about to take. Thus, scan 0 will be done at the first position, etc., with appropriate correction for positions for which there is a repeat count other than 1. Let's take the example shown in Figure 4, and refer to the four distinct positions shown as A,B,C,D. Also, assume that there isn't any fifth position which would be revealed by spinning the index spinner. Now, since B has a repeat count of 2, the list of positions is really A,B,B,C,D. The scans will be taken in the following order:

Scan #	Position
0	A
1	B
2	B
3	C
4	D
5	A
6	B
...	
32	B (32 mod 5 = 2)
40	A (32 mod 5 = 0)

As you can see from this example, the positioning is based on the scan number modulo the cycle length, with repeated positions counted separately.

One side effect of this way of specifying positions is that if there's a beam dump, the current scan will be written in its incomplete state, then the scan number will increment by one, so the program will go onto the next position, and you will have missed one. If you are doing a long series with the cycle repeating two or three times, then it's probable that you will get the missing point on the next cycle. The program automatically adds one to the number of scans to do if one is lost to a beam dump.

V. Uh-Oh!

The above procedures seem rather complex and counterintuitive when written down in a manual, but you will find as you use the beamline that there is a logic to it and it does make sense. There are a number of writeups available in the `10.3.2 Docs` directory (shortcut on the Desktop) which should be of assistance.

There is a troubleshooting guide on the desktop which gets updated every time a user manages to elicit a new symptom from the beamline. Read it if something is strange as your problem may have been seen before. There is a Trouble Call procedure which tells you whom to call and when, in case you get really stuck

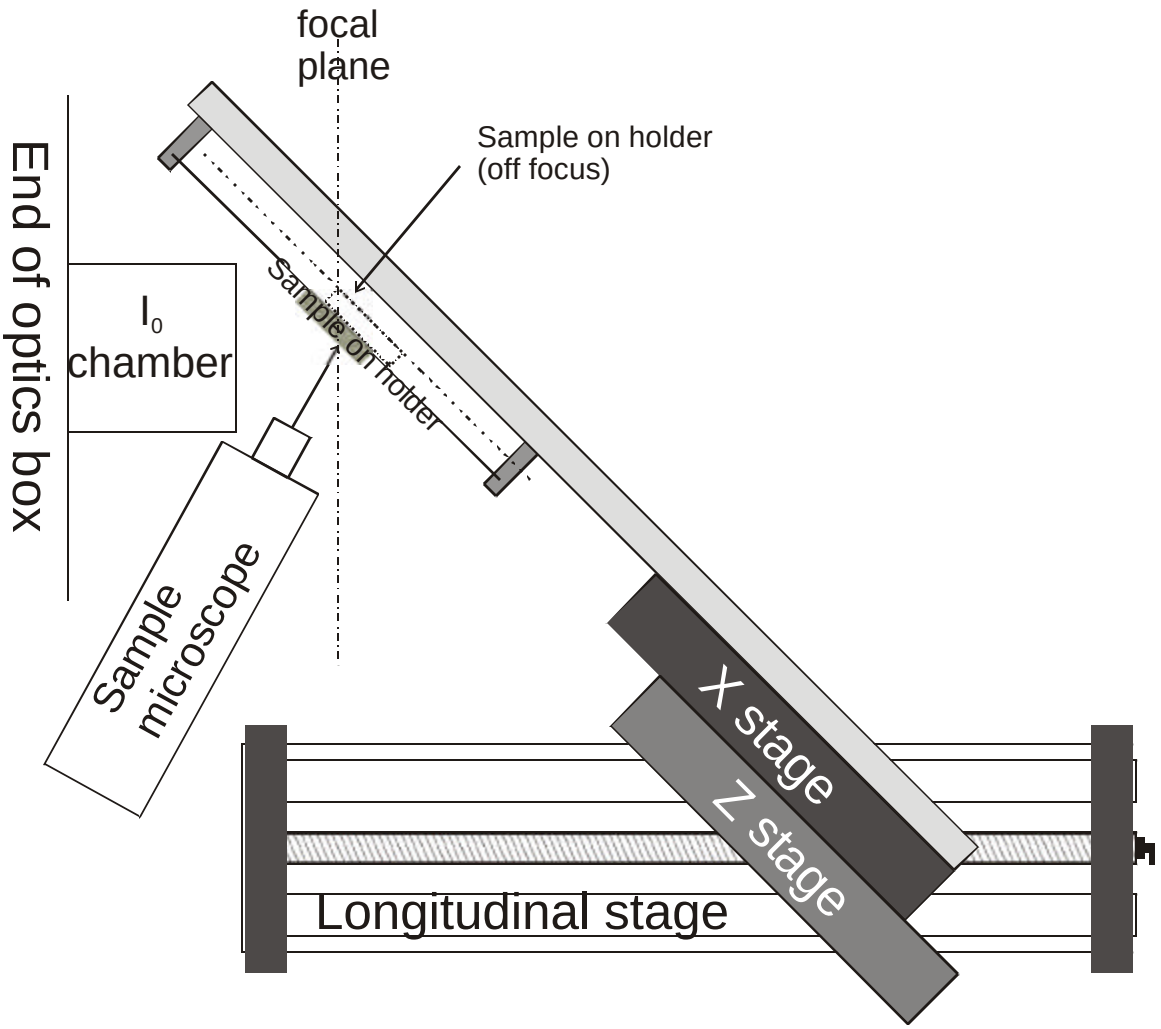

Figure 1. Overhead schematic view of the sample stage area showing the focal plane (dash-dotted line), the position of the sample when it's on focus, an off-focus position (in dotted lines) and the stages. The difference between the two positions lies in the adjustment of the longitudinal stage, which should be set so that the strike point of the beam on the sample is at the crosshair in the sample microscope.

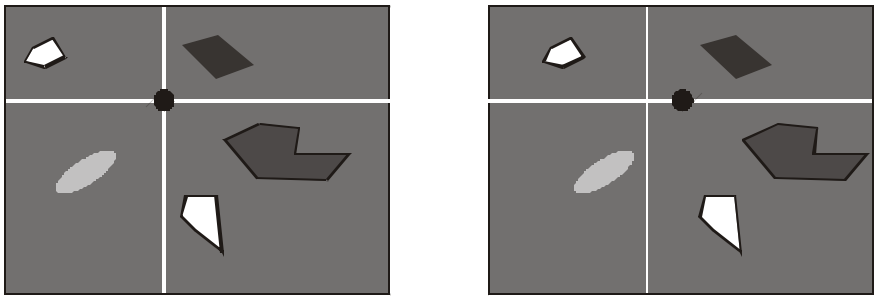

Figure 1b. The sample shown on- and off-focus. In both cases, the X-ray beam is hitting the center of the small, round, black grain. In the off-focus case, the crosshairs indicate a position to the left of where the beam actually hits because the sample is too far back and parallax creates an offset.

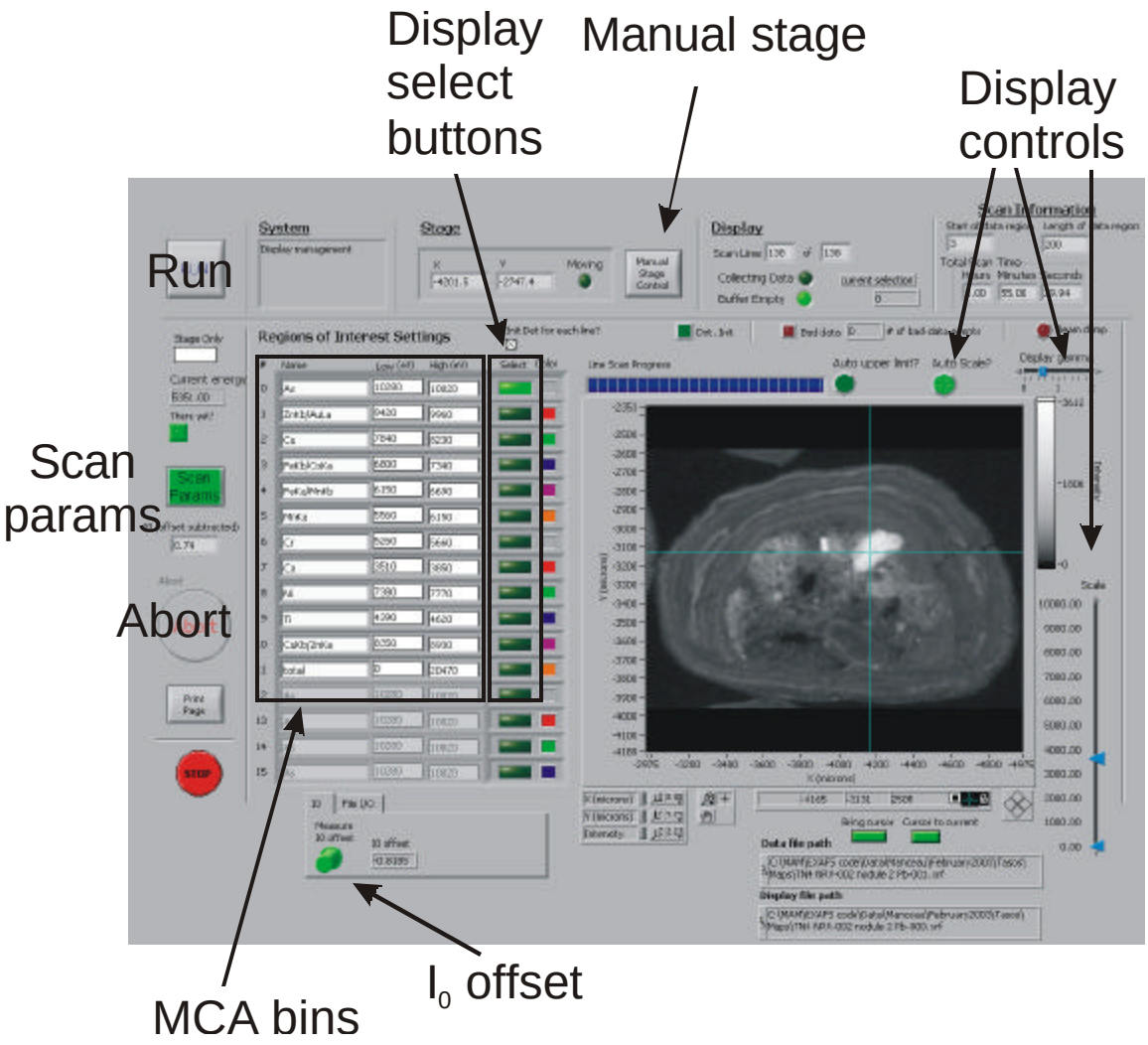

Figure 2. The XY mapping program with the important controls and indicators shown.

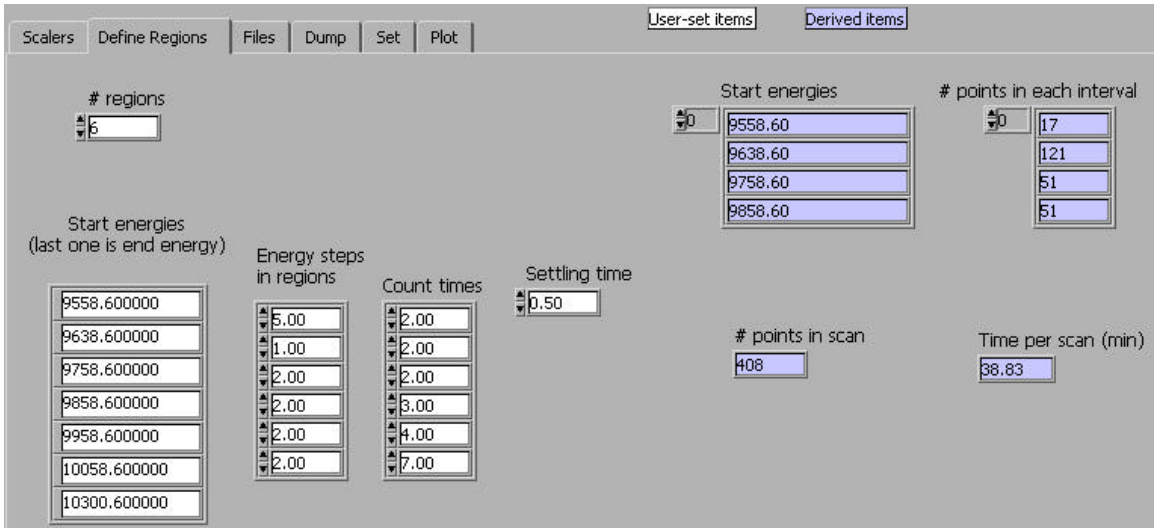

Figure 3a. This is the Define Regions screen of the scan editor. The Scalers tab is not normally used.

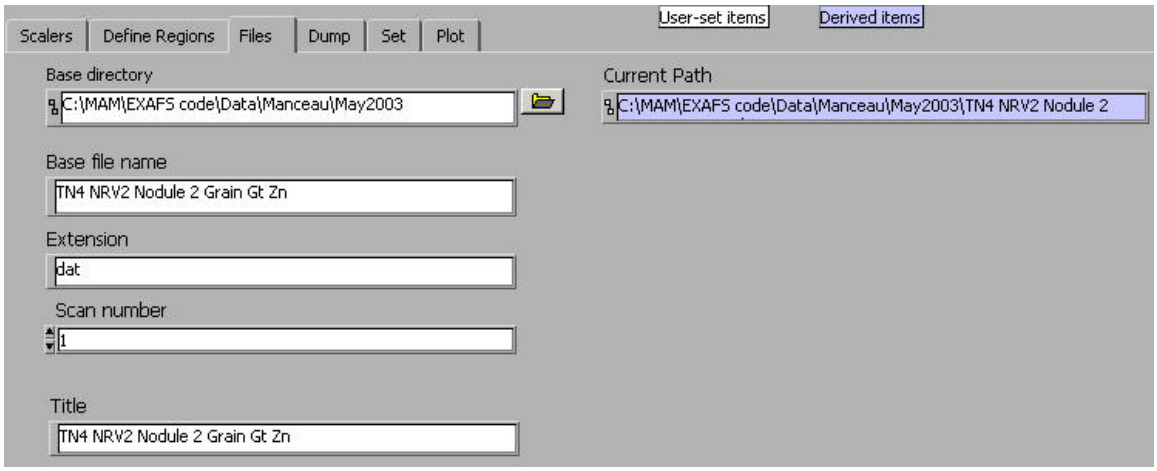

Figure 3b. The Files tab of the scan editor. The blue indicator is a little short to show the full path name: C:\MAM\EXAFS code\Data\Manceau\May2003\TN4...Zn001.dat.

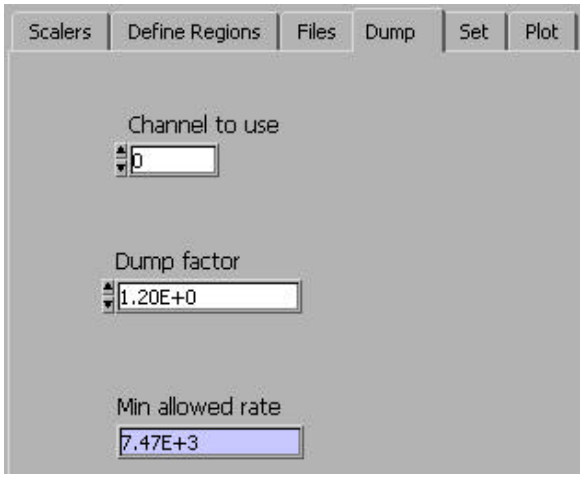

Figure 3c. The Dump screen. If the I_0 counts go below 7474 (0.0747V on the DVM), a beam-dump will be called.

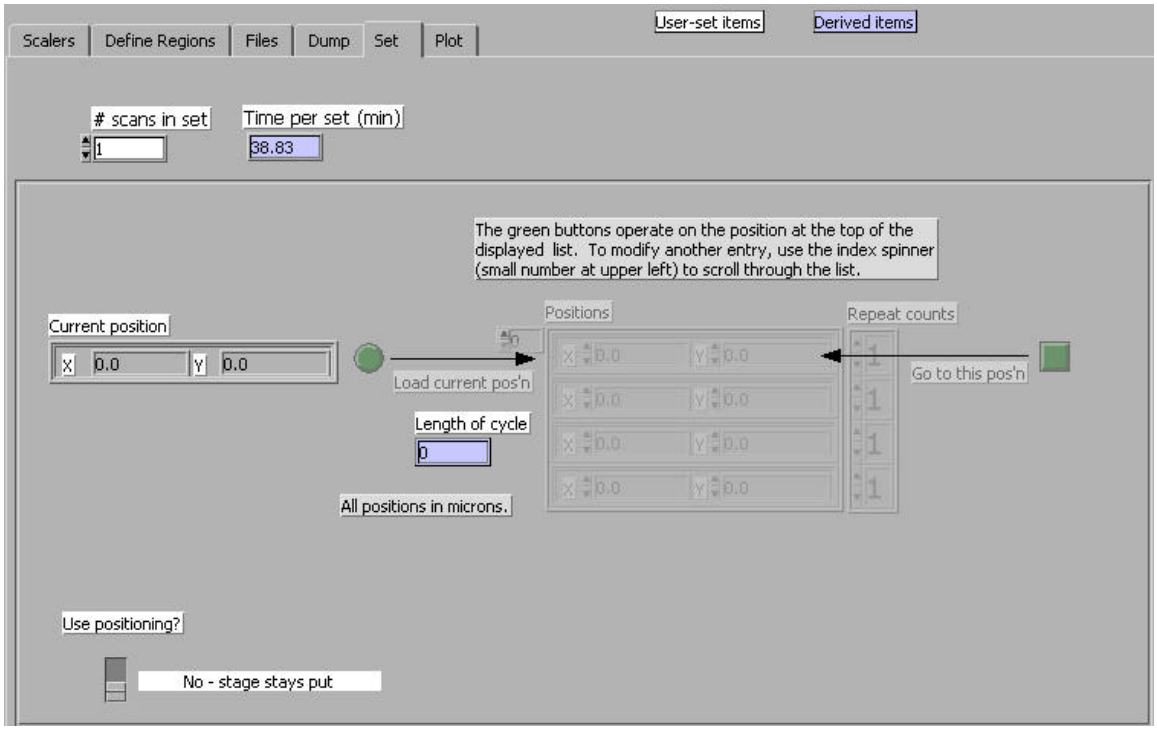

Figure 3d. The Set tab, with the multi-point feature disabled. The only control here is the number of scans.

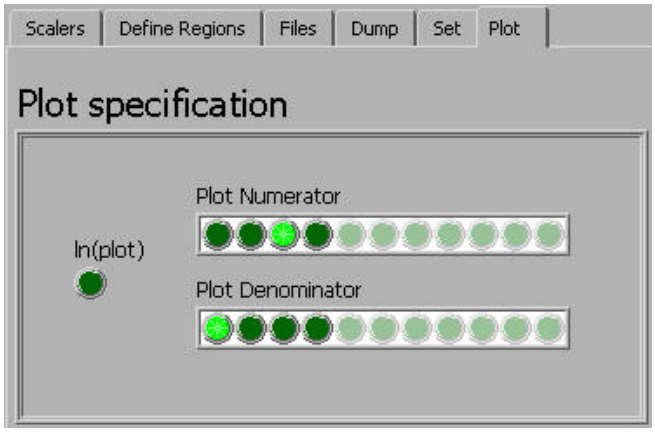

Figure 3e. The Plot tab, specifying that the quantity to plot is fluorescence divided by I0 (Scaler2/Scaler0).

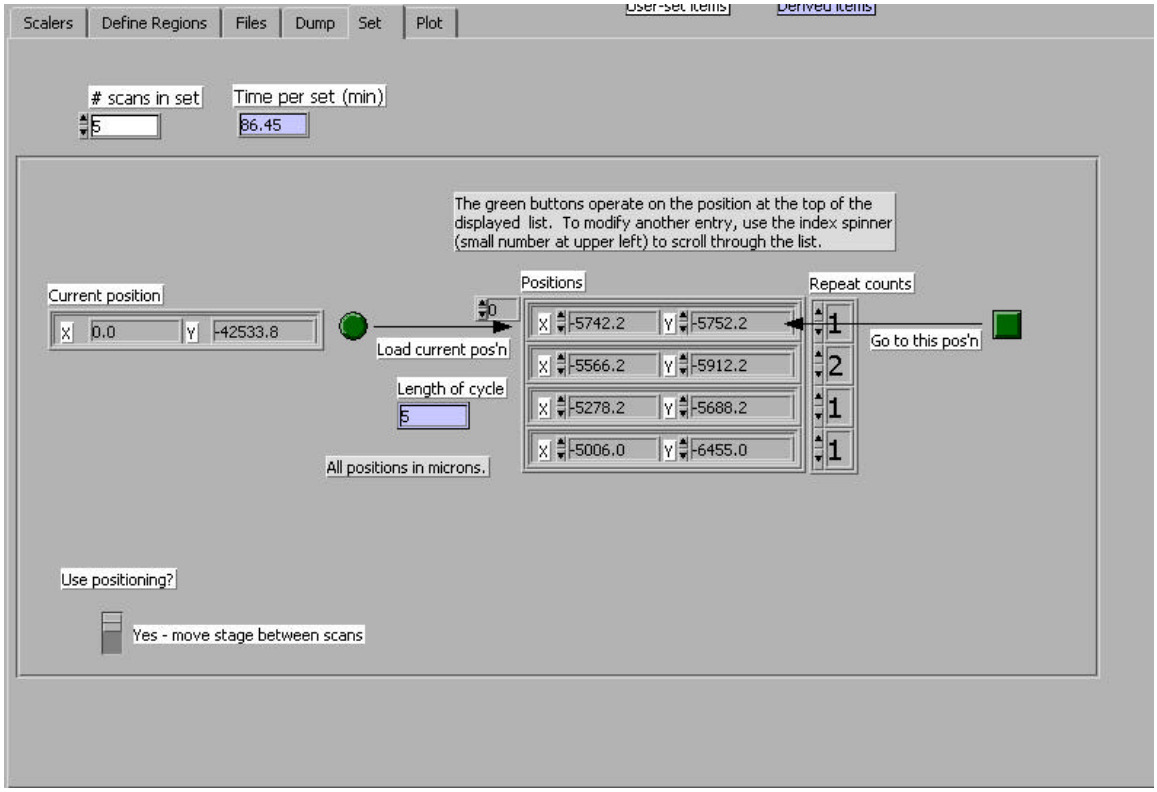

Figure 4. The Set tab showing the multipoint scan feature enabled.

Appendix A: Symptom-based troubleshooting guide

This is a partial list of trouble symptoms, some possible causes, and fixes.

Trouble contacts:

Matthew Marcus	x7604	510-981-1965 (H)
Sirine Fakra	x2975	510-524-5210 (H)

Please try the fixes listed below before calling.

Detection system:

1. No counts in MCA, mapping or EXAFS:

   You may be getting no beam. Check I0. If I0 is low or zero (after subtracting offset), see the Optics section.

   Detector HV may be off. This power supply is in the hutch. Check LN2 level before turning on.

   LN2 may have run dry. The auto-fill system control box is just outside the hutch, sitting on a yellow safety box. Its readout shows you how much (0-100) LN2 is left. If it's low, check:

   LN2 dewar empty? The level gauge can't be trusted - check the pressure instead.

   LN2 dewar valve closed? It tends to be left closed when the dewar is refilled.

   If LN2 ran dry, correct the problem and let the detector fill. Turn off the high voltage and wait 12-24hrs before turning it on and trying again.

2. Plenty of counts showing in MCA, but no edge jump recorded in fluorescence channel in EXAFS.

   When you edit the scaler map to switch to a new edge, you have to stop and restart the EXAFS program to make the program read in the new scaler map.

3. You find a hotspot in XRF mapping, but don't get any counts.

   Make sure you're above the edge.

   Make sure you did a move-to-crosshair in the Manual Stage Control.

4. You try to change the gain on I0 or transmission, but nothing happens.

   You have to turn the knob on the Keithley VI and then hit the blue Set button. Nothing happens until you hit that button.

Software - General

1. Buttons 'push' but nothing happens and they don't 'pop out' again when they should.

   When you invoke a program, it's in a dormant state. A small icon in the upper left will hold a white arrow if it's in that state. Push that arrow, and it will turn black, signifying that the program has started.

   The EXAFS and XY map programs have 'pop-up' subroutines (e.g. Measure and Scan Editor) which need to be Returned from before the rest of the program will work.

2. Data-taking computer completely locked up

   If you exit from all the stage programs, you can tickle a bug in the stage-control drivers (vendorware). This bug locks the computer. Hard-boot it. It's UXASES and it's in the half-rack labeled 'BRUKER AXS'. The login and password are posted at the line.

3. You stopped one of the programs, but another program thinks it's still running.

   If you use the small stop-sign icon to stop a program rather than its stop button, it doesn't have a chance to turn off the flag which says that it's running. Cure: You can start and stop the offending program properly, or you can use the desktop shortcut marked "No, it's NOT running!", which turns off that flag.

4. The data analysis computer shows a green screen without the icons to which you've become accustomed.

   That monitor is connected to three different computers via a KVM (Keyboard-Video-Mouse) switch. To switch to Computer 1, hit the sequence <Alt><Ctl><Shift>1<Enter>. Computer 1 is UXAS_DATA, the analysis computer. This is a similar situation to what obtains with the right (data-taking) screen, which is connected to two computers. The two-port KVM switch has a simpler sequence for switching - <Ctl><Ctl> toggles between the two machines.

5. You rebooted and now face a login/password screen.

Logins and passwords for the computers are posted above the left-hand screen. Each computer also has on its desktop a text file 'logins.txt' with this information. You can therefore look on one of the other computers for the 'key' to the one you're trying to open.

6. You try to print and nothing happens.

   This is a stupidity of our server system. All printing, even to the printers right at the beamline, goes through a server, XRAYS-1. If the connection is broken, it won't automatically re-establish itself. Go into Computers Near Me and double-click on XRays-1. It will want a login and password. Leave them blank and click OK. Printer icons should appear. Close the window and try again to print.

Software - EXAFS program:

1. Scan editor won't let you return

   You've entered something it doesn't understand or thinks makes no sense. Example: leaving the edge code off an edge-relative energy ("ge" instead of "gek").

2. Scan keeps aborting and restarting

   Dump level may be set incorrectly. If the dump level is too high, it will call a dump even if there isn't one.

   If the dump level is OK, then the channel for dump detection may have gotten set incorrectly. This should be set to 0 (default configuration) because that's the channel on which I0 comes in. This control is in the Scan Editor, under Dump.

3. EXAFS curve is very strange, but not due to 4) above.

   Make sure Differentiate switch is OFF.

   Make sure offsets were done correctly. Offset for detector channels should be 0. If not, then initialize detector and try again.

   Make sure your plot specification is correct (usually, 2/0).

4. Start button is grayed and disabled.

   Make sure MCA isn't running and that a fluorescence map isn't being taken. Indicators should pop up to tell you about these conditions.

5. Start button and mono-control buttons are grayed and disabled.

   If you change the mono energy from outside the program (say, using Motor Monitor) or have disabled the mono motor, the program gets confused and thinks there's a motion problem and that the mono is trying to get where it's told. Press the 'Mono really got there!' button on the Operations page to clear this.

   It sometimes happens that the communication with the server program on 1032_BL_CONTROLS is lost. In that case, no monochromator functions work. Stop and restart the EXAFS program to reconnect.

6. When doing a multi-point scan (automatic move), the stage doesn't go to the point you expected.

   The first position on the list is #0, the next #1, etc.
   The position it moves to when starting a scan is determined by the scan number:

   index = (scan #) mod (cycle length)

   Example: You have three positions, but have specified that the second one is repeated twice. The cycle is therefore

Scan 000	1st position
Scan 001	2nd position
Scan 002	2nd position
Scan 003	3rd position
...	
Scan 007	3rd position (7 mod 4 = 3, so it's like scan003)
Scan 008	1st position
...	

   Therefore, if you are restarting at other than scan 000, you may start from a position other than the first.

7. When doing a multi-point scan, you've missed one or two spots

   When there's a beam dump, the next scan after the one which was interrupted has a scan number which is three more than that of the last whole scan. You thus lose two positions.
   If the cycle runs to completion, the system knows that it has lost one, so you end up losing 1 or two positions, depending on repeat counts.

8. Energy reads 0 and maybe other things don't work

   Communication with the server on 1032_BL_CONTROLS was lost Stop and restart the EXAFS program. You may have to quit LabVIEW altogether to restore it.

Software - XY Mapping:

1. You keep getting 'Bad Data' errors.

   Beam dump level must be set correctly and beam must be present. (Yes, I've tried to map with the shutter closed; it doesn't work).

   Pixel size too small (<5um).

   Stage may be going too fast; increase dwell time.

2. Your map doesn't cover the area you think it should.
   You have to Return from the Manual Stage Control, then you have to hit Edit Scan to 'set' it.

   The map is always a little narrower than you specify because three points on the left and right are thrown out in order not to take points during which the stage was accelerating or decelerating.

3. Your map takes hours longer than you thought it would.
   Check the 'hours' indicator in the Scan Edit screen. It's easy to miss.

4. Make sure your spot size and scan pixel size differ by no more than a factor of 2, otherwise you waste either beam intensity or scan steps.

5. You added a channel to the Regions of Interest Settings area but no counts appear there.
   You have to stop and restart the program to make additional channels work.

6. Normalization doesn't seem to be working.
   Did you do an I0 offset?

7. One or two lines on your map are horizontally offset from the others.
   This is a known stage problem with no known cure except re-running the map. It's random.

8. The more-uniform areas of your map are speckled with defects consisting of one light pixel with a dark pixel immediately to its right.
   Another known stage problem. The stage 'catches' at random places instead of moving uniformly. You can try changing the

dwell time to change the motion speed and see if that helps, but there's no known cure.

Stage

1. Stage doesn't run.

   If stage stopped when you were moving it, you probably hit a limit. If the Unidex program is on the taskbar, then click on it and see if there are any error conditions showing. Press the Fault Ack button on the bottom of the screen, then press the Enable button on the left corresponding to whichever axis had the problem. Note that what the mapping program thinks of as Y is Z in the Unidex program. If this works, you don't need to reset or home anything.

   If the Unidex program isn't on, then start it using the icon which looks like

   ```
    ^
   /\ <--- blue
   ===
   \/ <--- black
    v
   ```

   Both axes will be disabled. Rack the stage back a couple of inches, enable X, then press its Home button (top of screen), then do the same for Z. This does the homing cycle. The stage will now move to its limits, so there had better be nothing in the way.

   Whenever the stage has been homed, it's in a position far from where you might want it. Pull up the manual stage control and hit Move to Start. This should move it to someplace reasonable. It sometimes happens that only one axis moves. In that case, hit it again and the other axis should go.

   If it still doesn't run, check the circuit breaker on the Aerotech controller on the right side of the electronics rack, under the notation "BR1032-06". This affects the Z axis.

   If the X-axis won't run and everything else seems OK, the problem may be the fuse inside the Aerotech controller. The Unidex program will complain about a velocity or position error when you try to move and will then disable the axis. Also, you will

be able to move the stage horizontally by hand and there will be no holding torque. This fuse is in the leftmost of the three unpluggable modules in the Aerotech driver box, next to the fan. Pull out the module, inspect the fuse and if need be, replace it. If the Unidex program is still on, it will know where it is and not need to be homed, though it will need to be enabled. This happens only if the stage hit something during a move, so be sure to clear any obstructions before trying the stage again.

2. You hit the Jog button in the Manual Stage Control and nothing happened.

   The buttons are a little flaky (some subtle timing problem) and don't always give you one and only one step at a time. Watch the current-position numbers to be sure.

Optics and X-rays

1. I0 jumps up and down 2-6% on a regular basis

   This is due to an EPU (probably #4) switching. You can't do much about it except try to get the normalization to work really well.

2. Beam is not well focused in the vertical direction at low energies.

   This is due to scatter off of M2 and you can't do much about it, either.

3. Normalization isn't good or data noisy

   Check offsets (dark counts).

   Make sure you're on a local max or min of count rates with respect to position.

   Try reducing the slit size if it isn't already somewhat down from full open.

   Try steering beam through roll slits with M1.

   If I0 < 1V, try raising the I0 gain, remembering to do offset again.

   Check to see that the I0 gas, if any, is appropriate. If

I0 was set up for Br with a rich Ar/N2 gas mixture, it will cause strange noise when running at lower energies. Similarly, if He is running and the energy is high, the I0 will be weak and noisy. The former error is harder to notice just by inspection of the signals.

If you're using a gas mixture, try switching to a single gas. The mixing panel sometimes doesn't produce a stable mixture.

4. Mono energy calibration drifts

   Check cooling water level in mono chiller.

   Make sure the hutch door doesn't stand open for a long time. Temperature fluctuations may affect calibration.

5. Data shows 0.1%-1% jumps at regularly-spaced energies (60eV interval at Cu edge; 80eV at Zn).

   This is a known problem with the monochromator and optics. What's happening is that a picomotor controls the gap between the two crystals to get a constant height offset in the mono. This motion is quantized in units of about 0.5um. For reasons unknown, when one of these steps occurs, the data can be affected, especially when the sample is highly inhomogeneous.

   The fix is to disable the motion during the scan. This is done as follows:

   1. Under the rack marked 'BR1032-04', between the 10.3.2 and 10.3.1 beampipes is a black box with switches and connectors. It's fairly well-hidden.

   2. On the upper right of the front panel of this box is a row of 4 switches, labeled 'Closed/Open-Loop Select'.

   3. Move to mid-scan and flip the leftmost (#1) switch to the up position. This will freeze the motion. The problem should now go away.

   4. IMPORTANT! Restore the switch position before moving to a different edge.

6. Fluorescence data shows strange dips or peaks in the scan

   Check the total counts (4th channel) and see if there's a big peak. If so, you may have a Bragg diffraction

peak being thrown into the detector. This can cause a deadtime-related dip by swamping one detector element with scattered light, or a peak in Fe or Cu by virtue of a beam hitting something in the detector. The latter case can be sneaky because the scattered beam causing the problem may not be directly detected.

Fix: Go to the energy at which this occurs. Use the MCA utility and look at each detector element. There will be one or two which show anomalously high counts in elastic, Fe or Cu bins. Go into the hutch and disconnect the BNC cable from the preamp whose number corresponds to the affected channel. The preamps are in two rows along the snout of the detector and each have a BNC and RJ connector on them. Then, check the MCA to see that the offending channel is truly disabled.
When doing deadtime correction, adjust the deadtime from 0.46us to 0.54us (1 detector disabled) or 0.64us(2 disabled).
*** Don't forget to reconnect it afterward! ***

7. Little or no beam at all.

Several possible causes for this. Check to see if there's been a beam dump (yes, that's obvious). Check to see if the Be-foil monitor or PIN diode (middle readout on DPMs) is zero. If so, it's possible the slits are closed down and you don't know it. Open them to 2000um each way and see if beam shows up. If it does, close them gradually until beam starts to be cut off. See if adjusting M1 roll and tweak do anything good. If M1 is centered, then the problem may be that one or both of the slits has an incorrect idea of where it is. Close down one of the slits until the beam just goes away. If the size shown on Motor Monitor isn't nearly zero, reset it to zero. You may have initially hit Reset instead of Move and cause this problem in the first place.

Suppose the monitor shows beam but you still don't get any in I0. The mono may be excessively detuned. If the Picomotor control box (black thing with knob on left and redpilot light on right) is set to channel 2 (mono tweak active), it seems to pick up some disturbance and move the tweak. Retune it and leave it set to an inactive channel (say, 1).

If the flux is good at one energy and dies at others, the

problem may be the mono servo switch mentioned in 6) above. If this switch is left in the up (freeze) position and you move to a very different energy, the beam out of the mono will move off the input acceptance of the KB pair and the output flux will drop. The cure is to put the switch down when changing edges or otherwise making a big energy move.

8. I0 pre-amp (in hutch) shows 'OVERLOAD'.
   If running He/N2 mix, you can't use the full 600V on I0 as it breaks down. Go down to 300V (connect to middle connector on battery box instead of end).

9. You're using the PIN diode for transmission and it has glitches
   It's made of single-crystal Si and has diffraction peaks. You can move these around by tilting and rotating, but you can't get rid of them altogether.
   Workaround: Use an ion chamber (there's a long one in the hutch somewhere) instead.

10. You're using an ion chamber for transmission and the signal is noisy.
    If the end windows aren't capped, slap some Kapton tape on them. You don't want the breezes blowing into it.

    If you're using Ar at energies below ~8keV, switch to N2. The absorption length at 7keV is about 3cm. You don't want all the beam absorbed in the first couple of cm because then much of the signal will come from places with nonuniform fields in the ion chamber.

    If you're not using Ar at energies above 10keV, switch.

11. All of a sudden, you see a rising or falling background
    If you're using a gas in I0 or another ion chamber, check the tank. You may have run dry.

Data and Analysis

1. Individual EXAFS scans look good, but average looks noisy or glitches don't normalize.
   The likely cause is that the sensitivity of the detection system, that is how much signal you get for a given I0 and absorbance, isn't the same between files. This has three common causes:
   1. The I0 gain was changed between scans.
   2. The detector was moved in or out between scans.

3. The concentration of the element of interest isn't the same from scan to scan. This is usually due to the spot moving on the sample, either intentionally (taking different spots to avoid radiation damage) or unintentionally.

The EXAFS Editor has an option for changing the gain on a channel, that is, multiplying the counts in a scaler by a constant. If I0 is much quieter than the fluorescence or transmission (usually the case)
then you want to do this adjustment on I0. For Cause 1, you obviously want to adjust the gain of the I0 channel so it's the same in all files. Thus, if some files were taken on the 1E-9 scale of the I0 current amp, and others at 1E-10, then use the Change Gain option to increase the I0 counts (scaler 0) in the 1E-9 files by 10x. This should make F/I0 or It/I0 come to about the same value in all files. For the other causes, if I0 is less noisy than the fluorescence or transmission (whichever you're using), then change the I0 gain so that the files all match in edge jump. It sounds a little odd to adjust I0 when it's the fluorescence channel which changed, but it's statistically sounder to do it this way if I0 is quieter, which it usually is.

A similar thing seems to happen if the time/point was changed for a region in one or more of the scans. I'm not sure why this is. Use the Jump Correct feature in the EXAFS Editor to 'fix' this.